

PATENT ABSTRACTS OF JAPAN

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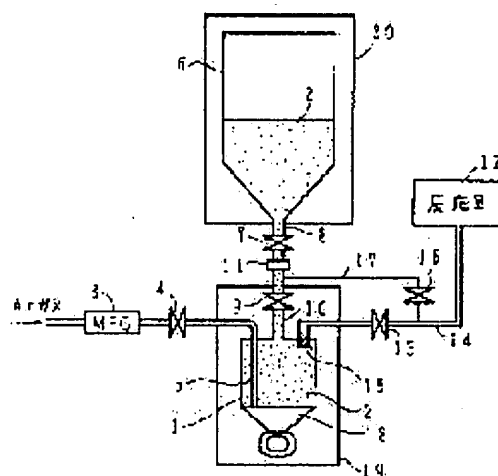
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(54) CVD DEVICE

(57)Abstract:

PURPOSE: To stabilize the gas supply rate of the CVD device by providing a solid gaseous raw material supplying system which sublimates the solid raw material, supplies this material to a reaction chamber and forms a thin film by chemical vapor growth.

CONSTITUTION: This CVD device has a 1st vessel 1 which houses the powdery or granular fluid solid raw material and the gas supplying system which sublimates this raw material and supplies the gaseous raw material to the reaction chamber. A 2nd vessel 6 which stores the solid raw material and replenishes this material to the 1st vessel is provided and 1st and 2d thermostatic chambers 19 and 20 for holding the 1st and 2nd vessels at prescribed temps. are provided. A low-frequency oscillator for agitating the solid raw material is installed to the 1st vessel 1. The raw material supply of the CVD device utilizing the solid raw material is stably executed in this way.



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DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to a CVD system and relates to the CVD system equipped with the solid-state material gas supply system which is made to sublimate a solid-state raw material, supplies a reaction chamber, and forms a thin film with chemical vapor deposition especially.

[0002]

[Description of the Prior Art] When forming a thin film using a CVD method, a start raw material can be mentioned to one of the most important factors. The CVD system whose raw material is a solid-state is examined with diversification of a technique in recent years. In the CVD method of aluminum expected as the next metallization technique for IC, alkylaluminum was conventionally used as a raw material. As a typical thing, triisobutylaluminum (TIBA), trimethylaluminum (TMA), a dimethyl aluminum hydride (DMAH), etc. are mentioned. Each of these matter is liquids in ordinary temperature ordinary pressure, and its vapor pressure is also low. In the CVD method of aluminum using these raw materials, if a substrate is heated at 230-300 degrees C, aluminum film deposits on a substrate by the pyrolysis of the gas of the vaporized raw material.

[0003] However, it is reported that recently still lower substrate temperature, i.e., the raw material which can be deposited by aluminum film at about 100 degrees C, exists. This raw material is matter called trimethylamine ARAN (TMAA), and is in a solid condition in ordinary temperature. As a description of TMAA, it is that the deposit temperature of aluminum film is low, and that aluminum film with very few impurities is obtained. Moreover, as a fault of TMAA, since the deposit temperature of aluminum is low, when saved also in ordinary temperature for a long period of time, it is that decomposition advances. In order to prevent this, it is necessary to maintain a raw material (TMAA) at low temperature as much as possible.

[0004] Although it is necessary to make a raw material sublimate and to supply a reaction chamber when performing a CVD method using this solid-state raw material, in the former, the approach of performing bubbling used from the liquid raw material as a configuration of a gas supply system, and a similar configuration exist. Drawing 2 shows the configuration of the conventional gas supply system. In drawing 2, the solid-state raw material 31 is held into the container 32 for sublimation, and the container 32 is further held in the thermostat 33. The solid-state raw material 31 is TMAA, and this [its] is granular. In order to supply the gas which was made to sublimate the solid-state raw material 31, and was obtained to a reaction chamber, carrier gas is introduced into the pars basilaris ossis occipitalis of a container 32, i.e., the lower part of the solid-state raw material 31, through the DIP tube 34. The sublimated gas moves to a reaction chamber through the piping 35 arranged in the upper part. The aforementioned thermostat 33 sets the container 32 whole as temperature a little higher than ordinary temperature in order to promote or control sublimation.

[0005]

[Problem(s) to be Solved by the Invention] The following problem existed in the gas supply system of the conventional CVD system mentioned above. The amount of the solid-state raw material 31 in a

container 32 decreases as use continues [1st]. It sets solid-state raw material 31, and since the capacity produced in sublimation is proportional to the surface area of a solid-state raw material, the amount of the solid-state raw material 31 will follow on decreasing, and capacity will also decrease gradually. That is, the stable fixed gas supply volume is unmaintainable. TMAA which is the solid-state raw material 31 the 2nd is the matter unstable in comparison also in ordinary temperature. Since it constitutes from an equipment configuration shown by drawing 2 so that the temperature up of all the solid-state raw materials 31 in a container 32 may be carried out using a thermostat 33, it becomes easy to produce disassembly of a solid-state raw material within a container 32, and in connection with the passage of time, gas supply volume decreases and the same problem as the above occurs.

[0006] In the CVD system equipped with the gas supply system of the conventional bubbler mold for the above-mentioned fault, membrane formation stabilized over the long period of time cannot be performed. Moreover, in order to perform stable membrane formation, there was fault of requiring complicated actuation of changing gradually the temperature of a container 32 and the flow rate of carrier gas according to the amount of sublimation.

[0007] In the CVD system using a solid-state raw material, the amount of supply of the gas sublimated over the long period of time is stabilized by the purpose of this invention, it is held uniformly, and is to offer the CVD system with which disassembly of the solid-state raw material in a hold container was equipped with very few solid-state material gas supply systems.

[0008]

[Means for Solving the Problem] By making said solid-state raw material in the 1st container which holds the solid-state raw material which has a fluidity, and this 1st container sublimate, in the CVD system equipped with the gas supply system which supplies material gas to a reaction chamber, the CVD system concerning this invention keeps a solid-state raw material, and it is constituted so that the 2nd container with which a solid-state raw material is supplied to the 1st container may be prepared. In said configuration, the 1st thermostat which holds the 1st container to predetermined temperature, and the 2nd thermostat which holds the 2nd container to predetermined temperature are prepared preferably, and whenever [every place constant temperature / of the 1st and 2nd thermostats] is set as different temperature. In each aforementioned configuration, the audio frequency oscillator for agitating a solid-state raw material in the 1st container is attached still more preferably.

[0009]

[Function] Since the large-sized container for the raw material storage for supplying this a solid-state raw material with the CVD system by this invention to the container for raw material sublimation was prepared, over a long period of time, the raw material level of the 1st container for sublimation can be maintained on predetermined level, and it becomes possible to hold the fixed amount of supply of gas to stability. Moreover, since it can be set as the temperature which was suitable for sublimation with the container for sublimation at suitable temperature, i.e., the container for storage, in each solid-state raw material with low temperature since a thermostat is independently prepared to each container and it enabled it to control the laying temperature independently, degradation of a raw material and decomposition can be prevented. In what was constituted so that it might have an audio frequency oscillator, the raw material for sublimation is fully diffused and a sublimation operation can be promoted.

[0010]

[Example] Below, the example of this invention is explained based on an accompanying drawing.

Drawing 1 shows one example of this invention. In drawing 1, 1 is a container which holds the solid-state raw material 2, and the solid-state raw material 2 in a container 1 is sublimated, and serves as gas. As an example of the solid-state raw material 2, it is granular TMAA. It is powder and the solid-state raw material 2 is granular or a thing which has a fluidity. A massflow controller 3 and the DIP tube 5 equipped with the adjustable valve 4 are connected to a container 1. The DIP tube 5 is inserted from the upper part of a container 1, and it extends and it is arranged until a point results near the pars basilaris ossis occipitalis. Therefore, the point of the DIP tube 5 is arranged at the lower part of the solid-state raw material 2. The argon (Ar) gas which is carrier gas is supplied with the DIP tube 5.

[0011] The solid-state raw material 2 is held and kept, and other containers 6 for filling up the solid-state raw material 2 according to the amount of the solid-state raw material 2 in a container 1 are arranged above the container 1 for sublimation. The container 6 has a bigger capacity than a container 1. For example, the capacity of 30 cc and a container 6 of the capacity of a container 1 is 1200 cc. The piping 8 which has a bulb 7, and the piping 10 which has a bulb 9 are formed in each of the lower part of a container 6, and the upper part of a container 1, and each piping 8 and 10 is connected to it with the joint 11. Piping 8 and 10 is using tubing of for example, the diameter of 1/4 inch.

[0012] 12 is the reaction chamber of a CVD system and a thin film is formed by vapor growth inside. A container 1 and a reaction chamber 12 are connected for the piping 14 equipped with the bulb 13. The gas which occurred with the container 1 is supplied to a reaction chamber 12 through piping 14. Moreover, the filter 15 was installed in opening by the side of the container 1 of piping 14, and it has prevented to it that the solid-state raw material 2 in a container 1 disperses in piping 14. Furthermore, between a bulb 9, the pars intermedia of a joint 11, and reaction chambers 12 and the pars intermedia of a bulb 13 is connected for the piping 17 equipped with the bulb 16.

[0013] The audio frequency oscillator 18 for agitating the solid-state raw material 2 held in the interior is arranged by the outside lower part in a container 1. This audio frequency oscillator 18 can give a several Hz - about 20Hz acoustic wave to the whole container 1.

[0014] The thermostat 19 only for these containers is formed, and the container 1 for sublimation is held in the interior of this thermostat 19. Moreover, the thermostat 20 of dedication is formed also in the container 6 with which keeps a raw material and a container 1 is supplemented suitably, and the whole container is held in the interior of a thermostat 20. It is possible to have two thermostats in 19 and for 20 to have the temperature controlled separately, respectively. Illustration of a control section is omitted in drawing 1 R> 1.

[0015] Next, an operation of the gas installation in the CVD system which has a gas supply system like the above is explained. Before operating a CVD system first, the container 6 for raw material storage with which it filled up with the solid-state raw material 2 is connected with a container 1 through a joint 11. From a container 6, the solid-state raw material 2 is moved to the container 1 for sublimation through piping 8 and 10. A container 1 is in the state of the sky at first. By migration of this solid-state raw material 2, at first, bulbs 4, 7, and 9 are closed, and where bulbs 13 and 16 are opened, the interior of a container 1 and piping between bulbs 7 and 9 is exhausted using the exhaustor (not shown) attached to the reaction chamber 12 of a CVD system. Then, by closing bulbs 13 and 16 and opening bulbs 7 and 9, the particulate-solid raw material 2 in a container 6 falls in a container 1, and a container 1 is filled up with the solid-state raw material 2. The container 6 for raw material storage is [about] always by between until an internal raw material is exhausted, and the thermostat 20. -It is cooled by about 15 degrees C.

[0016] Next, the gas for thin film production is supplied from a container 1 through piping 14 to a reaction chamber 12. Bulbs 7 and 9 are maintained by the condition of having opened. Furthermore, in order to supply gas, with a thermostat 19, the temperature of a container 1 is held at about 30 degrees C, and power is supplied to an audio frequency oscillator 18, and the solid-state raw material 2 held in the interior is agitated. In order to enable it to supply gas, the bulb 13 of piping 14 is opened. Next, a bulb 4 is opened and the argon gas in which control of flow was performed with the massflow controller 3 is introduced in a container 1 through the DIP tube 5. The solid-state raw material 2 is sublimated by installation of argon gas, and the gas which occurred is supplied to a reaction chamber 12 through piping 14. Although the solid-state raw material 2 in a container 1 is consumed by sublimation and the amount decreases, since it connects with the container 6 for storage for piping 8 and 10, a container 1 is continuously supplemented with a solid-state raw material.

[0017] Like the above, it can carry out and the material gas which was stabilized with sufficient repeatability and sublimated to the reaction chamber 12 can be supplied over a long period of time. If the solid-state raw material 2 in a container 1 and a container 6 is consumed, the amount of a solid-state raw material decreases and the level becomes less than a bulb 9, the flow rate of sublimation gas will begin to decrease gradually. When this reduction is detected, it will be necessary to newly refill up a

container 1 with a solid-state raw material. Then, it considers as the condition of having closed bulbs 7, 9, and 16, the container 6 for raw material storage used as empty is taken out, and other containers 6 for raw material storage with which it filled up with the raw material are set. Next, only a bulb 16 is opened and the piping part of a joint 11 is exhausted. After this exhaust air is completed, bulbs 7 and 9 are opened and a container 1 is filled up with the solid-state raw material 2. By the above, it is stabilized continuously and material gas can be supplied from a container 1 to a reaction chamber 12.

[0018] In addition, although the location of an audio frequency oscillator 18 was fixed and used in said example which used TMAA, the effectiveness which agitates the solid-state raw material 2 in a container 1 can be heightened as occasion demands by moving the location of an audio frequency oscillator 18 in the direction of the upper and lower sides or width periodically.

[0019] Moreover, although considered as the equipment configuration which exchanges the container 6 for storage in said example, it is also possible to make it the configuration which supplements a container 6 with a raw material. Furthermore, the configuration of the detection equipment of arbitration is employable as detection of whether the level of the solid-state raw material 2 in a container 1 became lower than a bulb 9.

[0020]

[Effect of the Invention] According to this invention, during a very long period, it continues, and it is stabilized and the gas sublimated from the solid-state raw material can be supplied by the constant rate by the above explanation so that clearly. Since the thermostat which can set up temperature independently of each of the container for raw material sublimation and the container for raw material storage was prepared, a solid-state raw material can be set as suitable temperature, and degradation and decomposition of a raw material can be prevented. Membrane formation stabilized with the CVD system by this can be performed.

[Translation done.]